(12) UK Patent Application (19) GB (11) 2 283 489 (13) A

(43) Date of A Publication 10.05.1995

(21) Application No 9421775.9

(22) Date of Filing 28.10.1994

(30) Priority Data
(31) 9322489 (32) 01.11.1993 (33) GB

(71) Applicant(s)
Delta Resins Limited

(Incorporated in the United Kingdom)

Moorfield House, Torkington Road, Hazel Grove, STOCKPORT, Cheshire, SK7 6NR, United Kingdom

(72) Inventor(s)
Andrew Seaborne

(74) Agent and/or Address for Service
Wilson Gunn M'Caw & Co.
26 Lockside, Marple, STOCKPORT, Cheshire, SK6 6BN, United Kingdom

(51) INT CL[6]
C08G 59/18 , C08F 2/46 , C08J 3/28

(52) UK CL (Edition N)
C3B BJ B1L3C B1L5X
U1S S1529 S1531 S1612 S1626 S2405

(56) Documents Cited
EP 0438123 A2

(58) Field of Search
UK CL (Edition N) C3B BJ , C3P PKB PKE PKN
INT CL[6] C08F 2/46 , C08G 59/18 , C08J 3/28 , C09J 5/06

(54) **Infrared curing of resins, resin systems and other catalysed systems**

(57) Resins, Resin Systems or other catalysed systems are cured by irradiation with infra red radiation at the temperature corresponding to the peak frequency characteristic of the spectrum of the curing reaction of the particular system. Irradiation may take place in an oven with electrical elements controllable to attain temperatures to the nearest 0.1°C, or catalytic gas burners to thus provide infra red radiation of the optimum frequency. An example of curing all epoxy resin system is given. Curing times in the order of ten minutes are achieved.

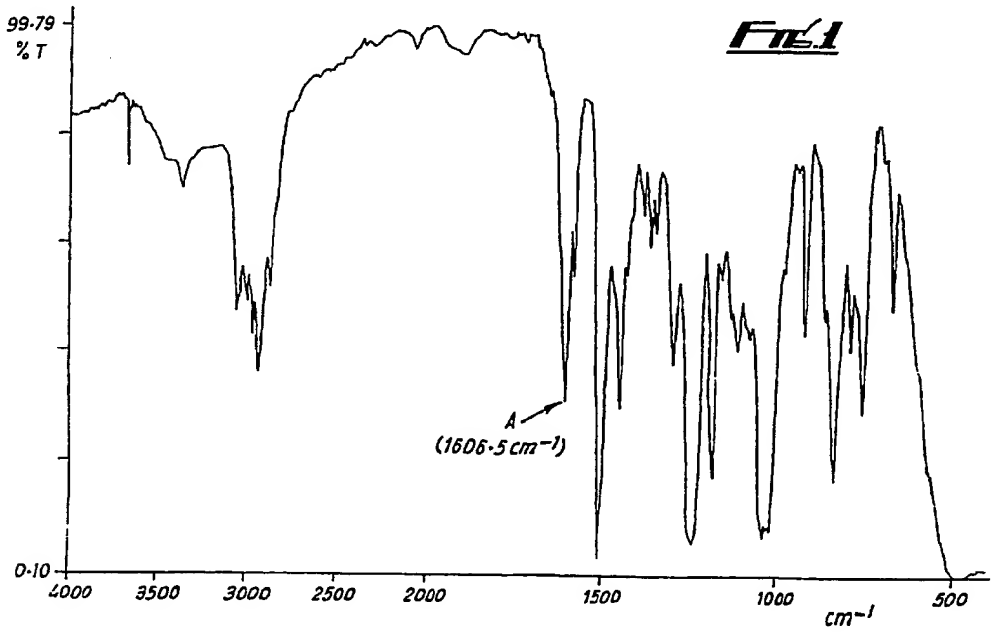

Fig.1

GB 2 283 489 A

At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.

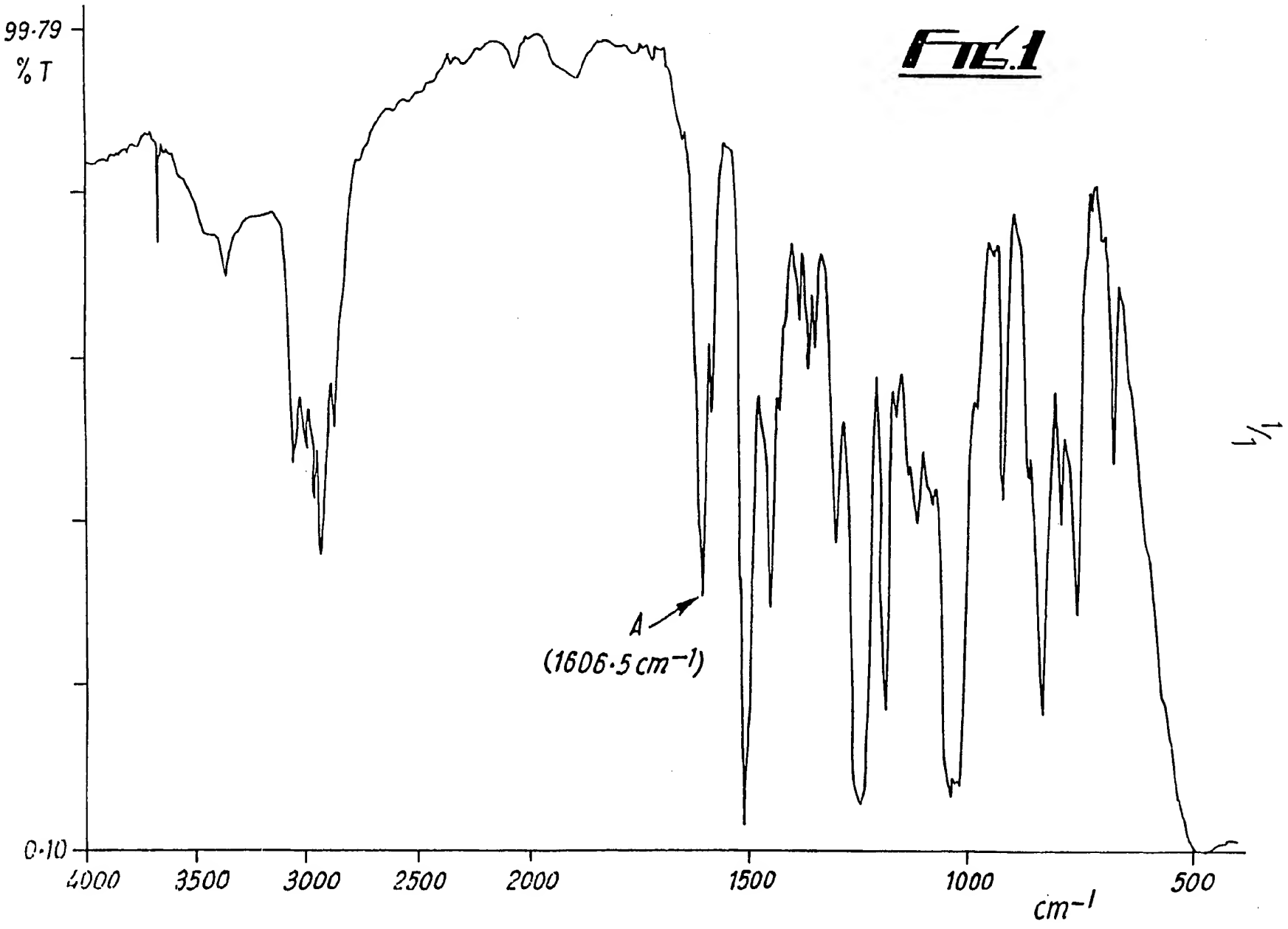
99·79
% T
0·10
4000
3500
3000
2500
2000
1500
1000
500
cm⁻¹
A
(1606·5 cm⁻¹)

FIG.1

2283489

CURING OF RESINS, RESIN-SYSTEMS AND OTHER CATALYSED SYSTEMS

This invention related to a method and apparatus for curing resins and resins systems and other catalysed systems using infra-red radiation.

In the current processes for curing resins and of resin systems by use of IR (infra-red) radiation, use is made of many forms of lamp arrays, ovens, etc. Often simple or complex catalysts are mixed into the resins to achieve an acceleration in curing time. Baths of glycol are also employed. Further, it is normal practice, in an effort to speed the curing process to use IR in excessive amounts, thereby generating considerable waste heat, and consuming excessive fuel or electricity. Much of the current technology used in the curing of resins and resin systems by IR, is too general and imprecise with resulting high costs and long curing times.

An object of the invention is to provide a method of curing resins and resin systems which will make possible considerably more efficient energy use, and shorter curing times than at present achieved, with resulting savings in cost and process time.

According to the invention, a method for curing resins, resin systems or other catalysed systems comprises irradiating the uncured resin or resin system with infra-red radiation which is characterised in that the radiation is emitted in a band having its peak at the peak frequency of the emission/absorption band of the curing reaction for the said resin or resin-system.

From another aspect, the invention provides apparatus for use in curing resins, resin systems or other catalysed systems by the method according to the invention, comprising infra-red

emitter means, the operating temperatures of such emitter means being selectable so as to provide infra-red emissions with peak intensity at the frequency of the emission/absorption band characteristic of the curing reaction for the said resin or resin-system.

The invention is based on the observation, that when examining the IR spectrum of a resin system, either with or without an admixture of one or more catalysts, it is found that only one peak in the IR spectrum is responsible for the occurrence of the curing reaction. Radiation is emitted at the corresponding frequency during the curing reaction, and conversely, absorptions of radiation at said frequency by the resin/resin system will promote the curing reaction. Broad-bank IR radiation outside the effective frequency band is thus largely waste heat, so that known IR heaters are corresponding inefficient in promoting the curing reaction.

It has been found experimentally that each resin or resin system has its own characteristic peak emission/absorption frequency, and consequently, the temperature of the IR emitting elements of any curing oven used in the method for curing resins or resin systems of more than one formulation, must be precisely variable in temperature. The optimum temperatures can be determined to 0.01 degrees centigrade and heaters should preferably be adjustable with the same precision. The method can also be used to overcome attractive forces such as Van der Waals forces, hydrogen bonding, or electrostatic charges, mainly in drying operations.

Control may be electro-mechanical, by means of a variable resistance or potentiometer in the electric supply circuit, or electronic using thyristor controls.

The peak frequency can be accurately translated into the optimum surface temperature of the heating elements and the electrical supply controlled to maintain the elements at the precise indicated surface temperature.

The attainment of the correct surface temperature of an element is determined by the wattage density at the surface. A calibration graph has been made from which the correct surface wattage density can be determined very accurately. As a consequence, it is now possible to design a heating element that simply needs to be plugged into the electricity mains in order to work properly. That is, no expensive, fragile and time consuming electronic controllers are required. Because the wattage density can be determined precisely and the surface temperature can be controlled by the density to remain continuously at the correct heat, the element design is much cheaper and very much more reliable than these at present in use.

Another heat source which may be suitable is a curing oven with one or more catalytic gas burners, which can be precisely controlled and burn without flame, emitting energy on a relatively narrow band of the I.R. spectrum. Such burners are thus very suitable for use in the method according to the invention.

The method of temperature control according to the invention enables an element to be fully energised, at the correct temperature throughout the curing period. If an element is working at 100% of its power, then it is operating more effectively than a more powerful element which is using say 80% of its capability. By use of the method of the invention, savings are obtained in both the amount of heat used, and in the cost of the heating elements. The invention makes possible a reduction in curing times as compared with these obtainable with

current methods, eg, from several hours to ± 10 minutes. As a result there are significant savings in energy costs, and increase in the quantity of resin which can be cured in a given working period.

It is also possible to use reduced amounts of catalyst and/or less aggressive catalysts may be used in resin systems to promote curing, this saves on the cost of catalyst and increases the pot life of the resin giving greater flexibility to production scheduling, and scorch-damage due to excessive heat is much reduced. Weaker catalysts are also safer to handle and less harmful.

The reduction in energy and catalyst use will also enable environmental improvements, both in the wider sense, and in the place of use.

The process is applicable to monomers, resin systems, paints, varnishes and plastics, where curing by IR is employed, both catalysed and non-catalysed resin systems can be handled, as laminating, bending, shrink wrapping and glass reinforced plastics, softening of plastics and thermoplastics for forming. Further possible applications are in drying or heat-treating textiles without damaging fibres, and in curing non-resin catalysed systems such as concrete mixes.

A preferred method of the invention will now be described with reference to an example, and the accompanying drawing, which is a spectrogram trace of a catalysed resin system used in the example.

Example

A standard epoxy resin system, catalysed by a cycloaliphatic amine is to be cured by the method of the present invention.

The catalytic action of the amine operates by breaking the amine into free radials, which then propagate the cross-linking or curing of the resins. The radicals are formed when the amine absorbs a particular amount of energy, which is provided by a particular wavelength of infra-red. Chemical data shows that this occurs between the wavelengths of 1550 to 1650cm $^{-1}$. The IR spectrum for this resin, when uncured, but with the catalyst added, shows a peak at 1606.5cm $^{-1}$ (See Fig 1, which is a spectrogram trace of the system at wavelengths between 5 and 40 microns). The relevant peak is indicated on Fig 1 by arrow A.

Using Wien's Law:-

$$\lambda mT = C$$

where λ = wave length in microns = 6.25 microns

T = absolute temperature in °K,

C = is a constant = 2898

A temperature of 192° 7C is obtained as the optimum temperature to effect curing of the resin system.

The temperature of the heating elements of an electrically powered curing oven were then adjusted, by means of a potentiometer to 192° 7C.

The curing of the resin system was found to be complete after approximately 10 minutes.

Claims:-

1. A method for curing resins, resin systems, or other catalysed systems, comprising irradiating the uncured system with infra-red radiation which is characterised in that the radiation is emitted in a band having its peak at the peak frequency of the emission/absorbtion band for the curing reaction for the said system.

2. A method according to Claim 1, wherein the characteristic frequency of the specific resin or other system is first determined from a spectrum scan of the uncured system, and the corresponding temperature determined, using Wiens Law,

$$\lambda MT = C$$

where λ = wavelength in Microns,

T = absolute temperature in °K.

and C = is a constant = 2898

3. A method according to Claim 2 wherein heat generating means in a curing oven are controlled to be heated to precisely the temperature determined by the step of Claim 2.

4. A method according to Claim 3 wherein a potentiometer, or thyristor control is used to precisely control the temperature of one or more electrical elements, to the nearest 0.1°C.

5. A method according to Claim 3, wherein the heat generating means comprise one or more catalytic gas burners.

6. A method for curing resins, resin systems, or other catalysed systems substantially as hereinbefore described, and with reference to the example given.

7. Apparatus for use in curing resins, or resin systems, or other catalysed systems by the method according to Claim 1, comprising infra-red emitter means, the operating temperature of such emitter means being selectable so as to provide infra-red emissions with peak intensity at the peak frequency of the emission/absorbtion band characteristic of the curing reaction for the said resin, resin system or other catalysed system.

8. Apparatus according to Claim 7, comprising a curing oven, furnished with one or more electrical heating elements which can be controlled by a potentiometer, or thyristor means, to determine the precise temperature of said element or elements to the nearest 0.1°C.

9. Apparatus according to Claim 7, comprising a curing oven, furnished with one or more catalysed gas burners.

Patents Act 1977
Examiner's report to the Comptroller under Section 17
(The Search report)

8

Application number
GB 9421775.9

Relevant Technical Fields

(i) UK Cl (Ed.N) C3B; BJ,C3P; PKB,PKE, PKN

(ii) Int Cl (Ed.6) C08F 2/46, C08J 3/28, C09J 5/06, C08G 59 18

Search Examiner
A KERRY

Date of completion of Search
16 FEBRUARY 1995

Databases (see below)
(i) UK Patent Office collections of GB, EP, WO and US patent specifications.

(ii) ONLINE DATABASES: WPI, CLAIMS

Documents considered relevant following a search in respect of Claims :-
1 - 9

Categories of documents

X: Document indicating lack of novelty or of inventive step.

Y: Document indicating lack of inventive step if combined with one or more other documents of the same category.

A: Document indicating technological background and/or state of the art.

P: Document published on or after the declared priority date but before the filing date of the present application.

E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.

&: Member of the same patent family; corresponding document.

Category	Identity of document and relevant passages		Relevant to claim(s)
X	EP 0438123 A2	(SHOWA DENKO) see Claims 1, 12 - 17 Examples 8, 11, 14, 15, 17, 24, 26	1

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents)